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"RE\_AFFIRMED 1995" IS:7602-1975

# Indian Standard "KEAFFIRMED 1990" SPECIFICATION FOR "KEAFFIRMED 1990" LEAD CHROMATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

UDC 661.876.281:662.2



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001



## Indian Standard

## SPECIFICATION FOR LEAD CHROMATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

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## Indian Standard

## SPECIFICATION FOR LEAD CHROMATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

#### 0. FOREWORD

- **0.1** This Indian Standard was adopted by the Indian Standards Institution on 25 February 1975, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.
- 0.2 Lead chromate is a synthetic inorganic chemical widely used in the manufacture of delay detonators and in pyrotechnic formulations. The existing Indian Standard on the subject, namely, IS: 50-1967\* covers the material for use in paint industry and does not cater to the requirements of explosive and pyrotechnic industry. The manufacturers of detonators and pyrotechnic compositions have been experiencing difficulty in obtaining the material of suitable quality. This standard has been laid down with a view to helping lead chromate manufacturers and users to make and procure standard quality of lead chromate.
- **0.3** This standard contains clause **3.1** which calls for agreement between the purchaser and the supplier.
- **0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

#### 1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for lead chromate for use in explosive and pyrotechnic compositions.

<sup>\*</sup>Specification for lead and scarlet chromes ( first revision ). †Rules for rounding off numerical values ( revised ).

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#### 2. REQUIREMENTS

- 2.1 The material shall essentially consist of lead chromate in the form of deep yellow finely divided powder free from oil, lumps and extraneous impurities.
- 2.2 The material shall comply with the requirements laid down in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR LEAD CHROMATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Volatile matter, percent by mass, Max	0.05	A-2
ii)	Matter soluble in water, percent by mass, Max	0.2	A-3
iii)	Water soluble chlorides (as Cl), percent by mass, Max	0.005	A-4
iv)	Iron (as Fe), percent by mass,  Max	0.005	A-5
v)	Carbon compounds (as CO <sub>3</sub> ), percent by mass, Max	0.012	A-6
vi)	Chromate (as PbCrO <sub>4</sub> ), percent by mass, Min	97.0	A-7
vii)	Lead (as PbCrO <sub>4</sub> ), percent by mass, Min	97.0	A-8
viii)	Fineness (material retained on 45-micron IS sieve, wet sieving with water)	Nil	<b>A-</b> 9
ix)	Average particle size, microns (by air permeability method)	5-8	A-10
x)	pH ( of aqueous solution )	4-6	A-11

#### 3. PACKING AND MARKING

**3.1 Packing**—The material shall be suitably packed as agreed to between the purchaser and the supplier.

- 3.2 Marking The container shall be marked legibly and indelibly with the following information:
  - a) Name and description of the material;
  - b) Manufacturer's name and/or his recognized trade-mark; if any;
  - c) Year of manufacture;
  - d) Tare and net mass of the material; and
  - e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.
- 3.2.1 The containers may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

#### 4. SAMPLING

**4.1** The method of drawing representative samples of the material, the number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

#### APPENDIX A

(Clause 2.2)

# METHODS OF TEST FOR LEAD CHROMATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

### A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1960\*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of the analysis.

<sup>\*</sup>Specification for water, distilled quality (revised).

#### A-2. DETERMINATION OF VOLATILE MATTER

**A-2.1 Procedure** — Heat a flat-bottomed clean glass or aluminium dish with cover about 6 cm in diameter and 3 cm in depth in an oven at  $100 \pm 2^{\circ}$ C for half an hour. Cool it in a desiccator and weigh. Place about 10 g of the material in the dish, replace the cover and weigh accurately. Uncover the dish and put it in an oven maintained at  $100 \pm 2^{\circ}$ C for 2 hours. At the end of this period replace the cover, cool the dish and place in a desiccator to cool to room temperature and weigh. Repeat the operation till constant mass is obtained.

#### A-2.2 Calculation

Volatile matter, percent by mass 
$$=\frac{100 \times (M_2 - M_3)}{(M_2 - M_1)}$$

where

 $M_1 = \text{mass in g of the empty dish and cover,}$ 

 $M_2 = \text{mass in g of the dish and cover with the sample taken,}$  and

 $M_3$  = mass in g of the dish and cover with the sample after heating.

#### A-3. DETERMINATION OF MATTER SOLUBLE IN WATER

**A-3.1 Procedure** — Weigh accurately about 10 g of the material dried as prescribed under **A-2** and transfer it into a beaker. Wet it thoroughly with 95 percent by volume of ethyl alcohol or rectified spirit and then add 200 ml of water. Boil for 5 minutes. Cool the mixture to room temperature and transfer to a 250-ml volumetric flask. Make up to 250 ml with freshly boiled and cooled water. Shake and filter. Reject about 50 ml of the first portion of the filtrate. Take a 100-ml portion from the rest of the filtrate in a tared porcelain dish and evaporate to dryness on a water bath. Dry the residue so obtained to constant mass in an oven at  $100 \pm 2^{\circ}$ C.

#### A-3.2 Calculation

Water soluble matter, percent by mass = 
$$\frac{(M_2 - M_1) \times 2.5 \times 100}{M}$$

where

M =mass in g of the material taken for test,

 $M_1 = \text{mass in g of the empty dish, and}$ 

 $M_2 = \text{mass in g of the dish with the residue.}$ 

#### A-4. DETERMINATION OF WATER SOLUBLE CHLORIDES

#### A-4.1 Reagents

- A-4.1.1 Standard Silver Nitrate Solution 0.1 N.
- A-4.1.2 Potassium Chromate Solution 5 percent approximately.

A-4.2 Procedure—Weigh 50 g of the material and transfer it to a 500-ml conical flask. Add 200 ml of water and shake thoroughly. Add a small quantity of filter paper pulp and filter the mixture through a sintered glass crucible No. G4. Wash the residue with several portions of water and add the washings to the filtrate. Add 1 ml of potassium chromate solution to the filtrate and titrate with standard silver nitrate solution to a permanent faint blood red tinge. Run a blank simultaneously.

#### A-4.3 Calculation

Chlorides (as Cl), percent by mass = 
$$\frac{3.55 (V - B) N}{M}$$

where

V = volume in ml of standard silver nitrate solution used for the sample,

B =volume in ml of standard silver nitrate solution used for the blank,

 $\mathcal{N} = \text{normality of standard silver nitrate solution, and}$ 

M = mass in g of the sample taken for the test.

#### A-5. DETERMINATION OF IRON

A-5.0 Outline of the Method — The material is dissolved in dilute nitric acid, lead is precipitated and removed as lead sulphate, iron and chromium precipitated as hydroxides from which chromium hydroxide is separated by treatment with sodium peroxide. The iron hydroxide is dissolved in hydrochloric acid and iron is colorimetrically estimated.

#### A-5.1 Reagents

- **A-5.1.1** Dilute Nitric Acid 50 percent (v/v).
- A-5.1.2 Concentrated Sulphuric Acid See IS: 266-1961\*.
- A-5.1.3 Ammonium Chloride solid.
- **A-5.1.4** Ammonium Hydroxide 20 percent (w/w).
- A-5.1.5 Sodium Peroxide solid.
- A-5.1.6 Dilute Hydrochloric Acid—1:1 by volume.
- A-5.1.7 Thioglycollic Acid

<sup>\*</sup>Specification for sulphuric acid (revised).

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A-5.1.8 Standard Iron Solution (Ferrous) — Dissolve 3.511 g of ferrous ammonium sulphate [FeSO<sub>4</sub>.(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O] in water containing 40 ml of 1 molar sulphuric acid. Dilute to 500 ml with water and mix the solution well. Dilute 10 ml of this solution to 1 litre with water in a volumetric flask containing 40 ml of 1 molar sulphuric acid. One millilitre of this dilute solution is equivalent to 0.01 mg of iron (as Fe).

#### A-5.2 Procedure

- A-5.2.1 Take 1.0 g of the material, accurately weighed, in a 250-ml beaker. Dissolve the lead chromate in 25 ml of dilute nitric acid. Add 5 ml of concentrated sulphuric acid and heat on a hot plate till copious fumes of sulphuric acid are evolved. Cool the solution in the beaker and cautiously dilute with water. Filter off the precipitate on a sintered glass crucible No. G4 and wash the precipitate 3-4 times with water. the filtrate add 2 g of ammonium chloride and a slight excess of ammonium hydroxide. Heat to boiling for a few minutes and set aside for the precipitates to settle. Filter the precipitate using Whatman No. 41 filter paper or equivalent and wash the precipitate with 3-4 times with hot water. Transfer the filter paper containing the precipitate to a 500-ml beaker containing 100 ml of water. Add about 1 g of sodium peroxide in small portions to the cold solution, till oxidation of chromium salt to chromate is complete. Then heat the solution to decompose the excess of sodium peroxide. Filter the solution through a Whatman No. 41 filter paper or equivalent and wash the precipitate 3-4 times with hot water. When washing is complete, place the filteration funnel on to a 100-ml Nessler cylinder, pierce the filter paper and dissolve the precipitate of iron oxide with 20 ml of dilute hydrochloric acid, collecting the acid solution in the cylinder. Estimate the iron content on the solution as detailed below
- A-5.2.2 To the solution in the 100-ml Nessler cylinder add 4 drops of thioglycollic acid, mix and add ammonium hydroxide until just ammoniacal, then add 1 ml in excess. Make up the volume to the 100-ml mark. In a second Nessler cylinder take 5.0 ml of the standard iron solution and the same quantities of thioglycollic acid, ammonium hydroxide, but not the sample solution and make up to 100-ml mark.
- A-5.2.3 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the intensity of colour produced with the sample solution is not greater than that produced by the standard solution.

#### A-6. DETERMINATION OF CARBON COMPOUNDS

**A-6.0 Outline of the Method**—Carbon is determined by igniting the material in a stream of oxygen, absorbing the carbon dioxide produced in barium hydroxide and comparing the turbidity produced.

#### A-6.1 Reagents

- **A-6.1.1** Barium Chloride Solution 10 percent approximately.
- A-6.1.2 Dilute Ammonium Hydroxide 8 N.
- A-6.1.3 Standard Sodium Carbonate Solution 0.01 N.
- A-6.2 Procedure Ignite 4 g of the material in a combustion tube in a stream of carbon dioxide free air or oxygen and pass the evolved gases into a mixture of 100-ml of carbon dioxide-free water and 10 ml of dilute ammonium hydroxide. Add 10 ml of barium chloride solution and leave to stand for 2 minutes.
- A-6.3 The limits prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced is not greater than that produced by the addition of 10 ml of barium chloride solution to a mixture of 98 ml of carbon dioxide-free water, 10 ml of dilute ammonium hydroxide and 2 ml of sodium carbonate solution and allowing it to stand for 2 minutes.
- A-6.4 Both the test and standard shall be protected from atmospheric carbon dioxide.

#### A-7. DETERMINATION OF CHROMATE

**A-7.1 Outline of the Method** — The material is dissolved in dilute hydrochloric acid and reduced with an excess ferrous ammonium sulphate which is titrated against standard potassium dichromate solution using ferroin as indicator. From the quantity of ferrous ammonium sulphate used up, the chromate is calculated.

#### A-7.2 Reagents

- A-7.2.1 Dilute Hydrochloric Acid approximately 3 N.
- A-7.2.2 Sulphuric Acid-Phosphoric Acid Mixture Mix 75 ml of concentrated sulphuric acid with 75 ml of concentrated phosphoric acid. Add this mixture cautiously to 250 ml of water, cool and dilute to 500 ml.
- A-7.2.3 Ferroin Indicator Dissolve 0.150 g of ortho-phenanthroline and 0.07 g of ferrous sulphate (FeSO<sub>4.7</sub>H<sub>.</sub>O<sub>.</sub>) in water. Dilute the solution to 10 ml with water and mix well.
  - A-7.2.4 Standard Potassiu m Dichromate Solution 0.1 N.
- **A-7.2.5** Ferrous Ammonium Sulphate Solution 0.1 N. Dissolve 39.2 g of ferrous ammonium sulphate hexahydrate [FeSO<sub>4</sub> (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O] in freshly boiled water to which 100 ml of dilute sulphuric acid [50 percent (w/w)] have been added. Transfer the solution to a 1 000-ml graduated flask, dilute it to the mark with freshly boiled and cooled water and mix well.
- A-7.3 Procedure Weigh accurately 0.4 g lead chromate into a 500-ml conical flask. Add 200 ml of dilute hydrochloric acid and dissolve all the chromate. Heat the solution to approximately 70°C on a hot plate and add 10 ml of a mixture of concentrated sulphuric acid and phosphoric acid and stir for 10 minutes. Ensure that the sample is

completely dissolved. Add exactly 50 ml of ferrous ammonium sulphate solution. The sample will turn green indicating complete reduction of the chromate. Wait for one minute. Add 0.5 ml of ferroin indicator solution and titrate the excess of ferrous ammonium sulphate with standard potassium dichromate solution until the red colour changes to pale blue green.

A-7.3.1 Run a simultaneous blank test using the same quantities of the reagents as for the sample.

#### A-7.4 Calculation

Chromate (as PbCrO<sub>4</sub>), percent by mass = 
$$\frac{(V_1 - V_2) \times 10.77 \times N}{M}$$

where

 $V_1$  = volume in ml of standard potassium dichromate solution used for the blank,

 $V_2$  = volume in ml of standard potassium dichromate solution used for the sample,

 $\mathcal{N}$  = normality of the standard potassium dichromate solution, and

M =mass in g of the sample used for the test.

#### A-8. DETERMINATION OF LEAD

A-8.0 Outline of the Method — Total lead is determined by precipitating the lead as sulphide which is then oxidized to lead sulphate. This precipitate of lead sulphate is dissolved in ammonium acetate solution to remove any silica and barium sulphate formed along with it. From the amount of lead sulphate finally obtained, total lead in lead chromate is calculated as PbCrO<sub>4</sub>.

#### A-8.1 Reagents

**A-8.1.1** Concentrated Hydrochloric Acid — See IS: 265-1962\*.

**A-8.1.2** Rectified Spirit -- See IS: 323-1959†.

**A-8.1.3** Ammonium Hydroxide -20 percent (w/w).

A-8.1.4 Hydrogen Sulphide — gas, from Kipp's apparatus.

A-8.1.5 Hydrogen Sulphide Water — saturated solution.

A-8.1.6 Dilute Nitric Acid — approximately 4 N.

**A-8.1.7** Bromine

A-8.1.8 Concentrated Sulphuric Acid — See IS: 266-1961.

A-8.1.9 Ammonium Acetate Solution - saturated.

<sup>\*</sup>Specification for hydrochloric acid (revised).

<sup>†</sup>Specification for rectified spirit (revised).

Specification for sulphuric acid (revised).

#### A-8.2 Procedure

- A-8.2.1 Weigh accurately approximately 0.5 g of the sample dried as described under 6.1 of IS: 33-1963\* into a clean dry beaker. Add 100 ml of water, 15 ml of concentrated hydrochloric acid and 5 ml of rectified spirit. Cover the beaker and boil the contents gently to reduce the chromates, which is noted by a change in the colour of the solution to green. Dilute to 200 ml with water. If any insolubles remain, filter and wash the same thoroughly with hot water to collect all the traces of lead in the washings. Collect the filtrate and washings and add ammonium hydroxide solution slowly until the formation of a faint precipitate takes place. Add again 10 ml of concentrated hydrochloric acid. Pass a rapid stream of hydrogen sulphide for 10 to 15 minutes. Dilute this solution to 400 ml with water and again pass hydrogen sulphide gas till the precipitation is complete. Allow the precipitates to settle, filter and wash well with hydrogen sulphide water.
- A-8.2.2 Transfer all the precipitate from the filter paper to a beaker containing 25 ml of dilute nitric acid. Treat the filter paper with bromine, filter off and add the filtrate to the nitric acid solution in the beaker. Boil until all the lead sulphide has dissolved and cool. Add 10 ml of concentrated sulphuric acid, and evaporate by boiling on a hot plate until dense white fumes are evolved. Cool the contents of the beaker, add 50 ml of rectified spirit and 50 ml of water while stirring and allow the contents to settle for 4 hours. Filter the precipitate through a tared sintered glass (G No. 4) or Gooch crucible. Wash the precipitate first with a mixture of equal parts of rectified spirit and dilute sulphuric acid and then with rectified spirit alone, until it is free from sulphate ions. Dry the precipitate to constant mass.
- A-8.2.3 Treat the precipitate on the crucible with boiling ammonium acetate solution till all the lead sulphate is dissolved. Wash the precipitate with hot water until the filtrate is free from sulphate ions. Dry the precipitate, cool in a desiccator and weigh to constant mass.

#### A-8.3 Calculation

Lead (as PbCrO<sub>4</sub>), percent by mass = 
$$\frac{106.58 (M_1 - M_2)}{M}$$

where

 $M_1 = \text{mass in g of the crucible containing the precipitate}$  before treating with ammonium acetate solution,

 $M_2$  = mass in g of the crucible after treating with ammonium acetate solution, and

M = mass in g of the sample taken for the test.

<sup>\*</sup>Methods of test for dry pigments and extenders for paints (revised).

#### A-9. DETERMINATION OF FINENESS

- A-9.1 Place 100.0 g of the material on a 45-micron IS sieve and wash the material with large volumes of water, gently brushing the material with a soft camel hair brush till the washings are quite clear. Dry the sieve with the residue in an oven maintained at 100-102°C. Brush the dried residue into a tared scoop and find out the mass of the residue retained on 45-micron IS sieve.
- **A-9.2** The mass in g of the material retained on the sieve is the percentage of the material retained on 45-micron IS sieve.

# A-10. DETERMINATION OF AVERAGE PARTICLE SIZE AND SPECIFIC SURFACE (AIR PERMEABILITY METHOD)

- **A-10.1 Outline of the Method** The specific surface of the powder is first measured by the time of flow of a known volume of air through a compact bed of the material. The mathematical basis for the procedure is complex and to a certain extent empirical. The mean particle diameter is then calculated from its specific surface if the particle shape is known. For spherical particles the formula,  $X = 6 \div S$  is used, where, X = mean particle diameter in cm, and  $S = \text{specific surface in cm}^2/\text{cm}^3$ .
- A-10.2 Apparatus as shown in Fig. 1. \*It consists of a graduated glass U-tube manometer, a ground glass joint supporting a stainless steel test cell, a glass tube with stopcock leading to a rubber air aspirator bulb, all mounted on a wooden support stand forming one compact portable self-contained assembly. Behind the manometer is a stainless steel mirror to permit easier reading of the graduations of the manometer and to reduce parallex. The stainless steel test cell has an inner ledge which supports a stainless steel perforated disc. The test cell can also hold a stainless steel plunger used for compacting the sample bed. The manometer contains diesel oil of known density up to a height of 12 cm from the bottom of the U-tube.

#### A-10.2.1 Determination of Constant of the Test Cell

- **A-10.2.1.1** Determine the inside diameter of the cylindrical test cell by means of slide callipers and calculate the area of the bed of powder in  $cm^2(A)$ .
- **A-10.2.1.2** Determine the depth of the cell with the perforated disc and the two filter paper discs (that will be used later in the experiment) placed on the metal perforated disc. Subtract from the cell depth, the height of the stainless steel plunger from bottom end to collar, to obtain depth of the bed of powder in cm (L).

<sup>\*</sup>This apparatus is known as 'Blain air permeability fineness tester'.

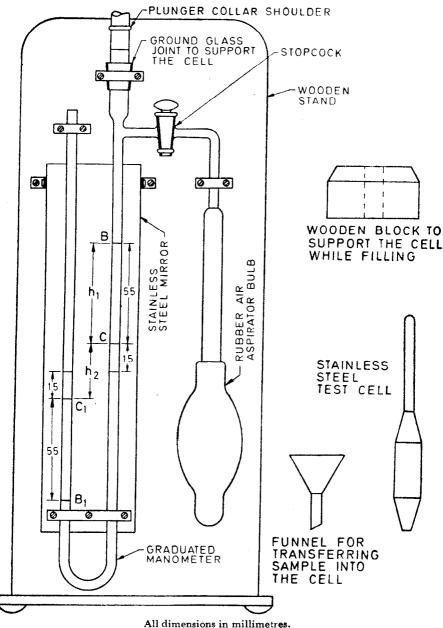


Fig. 1 Apparatus for the Determination of Average Particle Size — Air Permeability Fineness Tester

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**A-10.2.1.3** Determine the volume of manometer arm between marks B and C by filling from a burette. This volume (V) in ml will be equal to the volume of air passing through the bed in the experiment.

#### A-10.3 Procedure

- **A-10.3.1** Mix the whole sample well on a sheet of paper and make sure that its moisture content is within specification. Weigh out exactly 4.200 g of the sample of lead chromate on to a sheet of glazed paper. The quantity of the material taken is such that it gives a bed of standard porosity of 0.63 when pressed into the test cell (for calculation of porosity see **A-10.4.1**).
- **A-10.3.2** Place the stainless steel perforated disc in the clean dry stainless steel test cell, making sure that the disc sits properly on the inner ledge.
- **A-10.3.3** Cut two discs of filter paper exactly of the same diameter as the inside of the test cell using the punch provided and place one disc on top of the perforated disc.
- **A-10.3.4** Place the test cell in the wooden block usually provided with the apparatus and transfer the weighed lead chromate into the test cell using a funnel.
- **A-10.3.5** Consolidate and level the powder by tapping the cell. Place the second filter paper disc on top of the powder, insert the stainless steel plunger and push it down until the shoulder of the plunger rests on the lip of the test cell.
- **A-10.3.6** Withdraw the plunger from the test cell slowly in order to avoid formation of partial vacuum. Fix the test cell firmly into the ground glass joint.
- **A-10.3.7** Partially exhaust the air from the arm of the manometer through the glass stopcock and aspirator bulb until the level of the liquid in that arm comes well above the mark *B*.
- **A-10.3.8** Close the stopcock and note with a stopwatch the time required for the level of the liquid to fall from mark B to mark C.
- **A-10.3.9** Repeat the timing operation to give a total of 3 or 5 readings, 3 if the readings are constant and 5 if there is a fair range in the readings.
- **A-10.3.10** Calculate the average time in seconds required for the liquid to fall from B to C and determine the specific surface (surface area per unit volume of particle in  $cm^2/cm^3$ ) and particle size in cm as given in **A-10.4.1** and **A-10.4.2**.

#### A-10.4 Calculation

**A-10.4.1** Specific surface (S), cm<sup>2</sup>/cm<sup>3</sup> = 
$$\sqrt{\frac{g A p T}{K V N L}} \times \frac{e^3}{(1-e)^2}$$

where

g = acceleration due to gravity in cm/s<sup>2</sup>;

A =cross-sectional area of the cell in cm<sup>2</sup> which is also the area of bed of powder in cm<sup>2</sup>;

T = time of flow of air in seconds;

e = porosity, that is, volume of pore space per unit volume of bed, and is equal to:

l — (mass in g of the material)/density of powder volume in ml of the bed

(density of lead chromate is taken as 6.2 to 6.3 g/ml and shall be determined in each case),

K = a constant which has a value of 5;

V = total volume of air passing through the powder in ml and is equal to volume of the manometer arm between marks B and C;

N = viscosity of air in poises. This varies from 0.0001827 poise at 18°C to 0.000 195 8 poise at 40°C and is usually taken as 0.000 185 unless otherwise stated;

L = depth of powder bed in cm; and

P = mean effective pressure difference across the bed of powder, in g per cm² and is calculated from the commonly used equation:

$$H = \frac{H_1 - H_2}{\log_e (H_1/H_2)} \dots \dots (1)$$

where

H = mean effective head,

 $H_1$  = initial head, and

 $H_2 = \text{final head.}$ 

In terms of the apparatus shown in Fig. 1, the final pressure  $H_1d = (CC_1)d = H_1d$ , where d is the density of the oil in the manometer.

Initial pressure  $H_1d = (BC + CC_1 + C_1B_1)d = 2h_1d + h_2d$ . (B,  $B_1$ , C,  $C_1$  and  $h_2$  are same as in Fig. 1).

Substituting these values in equation 1, the mean effective pressure:

$$P = \frac{2h_1 d}{\log_e (1 + 2h_1/h_2)}$$

or in terms of common logarithm

$$P = \frac{0.8686 h_1 d}{\log_{10} (1 + 2h_1/h_2)}$$

Using diesel oil of density 0.857 3 g/ml at 15°C and a value of 55 mm for  $h_1$  and 30 mm for  $h_2$  as shown in Fig. 1, p works out to 6.12 g/cm<sup>2</sup>.

10.4.2 Particle Size — Calculate the mean particle size as follows:

Mean particle diameter in cm = 
$$\frac{6}{S}$$

where

 $S = \text{specific surface in cm}^2/\text{cm}^3$  as obtained in **A-10.4.1**.

#### A-11. DETERMINATION OF pH

**A-11.1 Procedure** — Weigh 5 g of the material and transfer it to a 500-ml beaker. Add 100 ml of freshly boiled and cooled water which is neutral to methyl orange indicator solution. Allow to stand for 30 minutes with occassional stirring. Filter, reject the first 50 ml of the filtrate and collect the remaining filtrate in a beaker. Determine the pH of the solution by means of a suitable pH meter using glass electrode.

#### APPENDIX B

(Clause 4.1)

# SAMPLING OF LEAD CHROMATE FOR EXPLOSIVE AND PYROTECHNIC COMPOSITIONS

#### **B-1. GENERAL REQUIREMENTS OF SAMPLING**

- **B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.
- B-1.1 Samples shall not be taken in an exposed place.
- **B-1.2** The sampling implement and the containers for samples shall be made of metal or opaque glass on which the material has no action. They shall be clean and dry.

- **B-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from adventitious contamination.
- **B-1.4** The sample containers shall be of such a size that they are almost, but not completely, filled with the sample.
- **B-1.5** The sample container shall be sealed air-tight after filling and marked with full identification particulars such as the date of sampling, the month and year of manufacture of the material, the name of the sample and any other relevant particulars of the consignment.
- **B-1.6** Samples shall be stored in such a manner as to avoid excessive variations of temperature.

#### B-2. SCALE OF SAMPLING

- **B-2.1 Lot** In a single consignment of the material all the containers of the same type and size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different type and size of containers, the containers belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.
- **B-2.1.1** For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2 given below.

TABLE 2 SCALE OF SAMPLING

LOT SIZE	SAMPLE SIZE
${\mathcal N}$	n
(1)	(2)
Up to 20	1
21 ,, 60	2
61 ,, 150	3
151 ,, 300	4
301 ,, 500	5
501 and above	6

**B-2.1.2** These containers shall be selected at random. In order to ensure randomness of selection, random sampling procedures given in IS: 4905-1968\* may be followed.

<sup>\*</sup>Methods for random sampling.

#### **R-3. PREPARATION OF TEST SAMPLE**

- **B-3.1** From each of containers selected, draw with an appropriate sampling instrument a small representative portion of the material approximately 100 g in mass.
- **B-3.2** Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly to form a composite sample of mass about 200 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for supplier and the third to be used as a referee sample.
- **B-3.3** The composite referee sample shall bear the seal of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

#### **R-4. NUMBER OF TESTS**

**B-4.1** Tests for the determination of all the characteristics mentioned in Table 1 shall be carried out on the composite sample.

#### **R-5. CRITERIA FOR CONFORMITY**

**B-5.1** For declaring the conformity of the lot to the requirements of this specification, the test result for each of the characteristics shall meet the corresponding values specified in Table 1.

#### INDIAN STANDARDS

ON

#### EXPLOSIVES AND RAW MATERIALS FOR EXPLOSIVES

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301-1963 438-1972	Potassium nitrate for explosives and pyrotechnic compositions (revised) Aluminium powder for explosives and pyrotechnic compositions (first
708-1970	Potassium chlorate, technical (first revision)

Red phosphorus 2012-1961

Magnesium powder for explosives and pyrotechnic compositions 2307-1962 Barium nitrate for explosives and pyrotechnic compositions 4396-1967

Ammonium nitrate for explosives 4668-1967

Lead thiocyanate for explosive and pyrotechnic compositions 5670-1970

Strontium nitrate for pyrotechnic compositions 5671-1970

Manganese dioxide for explosive and pyrotechnic compositions 5713-1970 Antimony sulphide for explosive and pyrotechnic compositions 5731-1970

6609 (Part I)-1972 Methods of test for commercial blasting explosives and accessories: Part I Gun powder

6609 (Part II/Sec 1)-1973 Methods of test for commercial blasting explosives and

accessories: Part II/Sec 1 Explosives, general 6609 (Part II/Sec 2)-1974 Methods of test for commercial blasting explosives and accessories: Part II/Sec 2 Explosives, permitted

6609 ( Part III )-1973 Methods of test for commercial blasting explosives and accessories: Part III Detonators, general and permitted

6609 (Part IV)-1972 Methods of test for commercial blasting explosives and accessories: Part IV Detonating fuses

6609 (Part V)-1972 Methods of test for commercial blasting explosives and accessories: Part V Safety fuses

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